

PATENT SPECIFICATION

(11) 1 396 757

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- (21) Application No. 47089/72 (22) Filed 12 Oct. 1972
 (31) Convention Application No. 2 150 872 (32) Filed 13 Oct. 1971 in
 (33) Germany (DT)
 (44) Complete Specification published 4 June 1975
 (51) INT CL² C08J 9/30
 (52) Index at acceptance
 C3C 123 151 354 358 452 503 521 543 546 548 553



(54) PROCESS FOR THE MANUFACTURE OF LATEX FOAM MOULDINGS

(71) We, CHEMISCHE WERKE HULS AKTIENGESSELLSCHAFT, a German Company, of 4370 Marl, Germany, do hereby declare the invention, for which we pray that a Patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following Statement:—

The present invention relates to the manufacture of latex foam mouldings from styrene-butadiene polymer latices which have not undergone any freeze agglomeration, by foaming the latex, filling the foam in non-gelled state into a mould which is at 40 to 70° C. and vulcanising.

In the manufacture of latex foam by the Dunlop process, the latex is filled as necessary with vulcanisation agent, sensitizer and foaming agent and, if desired, filler, and is brought to the desired specific gravity by whipping air into it. When it has reached the appropriate degree of foaming, the gelling agent is mixed in and the foam is filled into the mould. The mould is closed and is immediately, or after the foam has gelled, introduced into a heating tunnel for vulcanisation. After vulcanisation, the moulding is removed from the mould, washed and dried.

However, this procedure suffers from the disadvantage that when using latices which have not undergone a freeze agglomeration, surface flaws are produced on the mouldings if hot moulds are used. These flaws are described as "blistering". They are pea-sized blisters, the surface of which is covered by a thin skin. When using most latices it has hitherto been necessary that the moulds, which after removal of the mouldings and spraying with release agents are still at a temperature of 50 to 70° C., should be cooled to below 35° C. just for introducing the foam, and then again heated to 100° C.

This procedure is economically unfavourable and causes a distinct increase in the energy consumption coupled with a reduction in the capacity of the processing installations,

since considerable time is required to cool and heat the moulds, the walls of which are in part very thick. Cooling can—at least in the final phase—only be effected with air, since the moulds must be internally completely dry again when they are filled.

It was the task of the present invention to discover a possible way of manufacturing latex foam mouldings which are free of the surface flaws described from styrene-butadiene polymer latices which have not undergone any freeze agglomeration.

According to the present invention there is provided a process for the manufacture of a latex foam moulding from a styrene-butadiene polymer latex in which the styrene:butadiene weight ratio is from 1:4 to 1:1 and which has not undergone any freeze agglomeration, by foaming the latex, filling the foam in un-gelled state into a mould which is at a temperature of from 40 to 70° C. and vulcanising, wherein the foamed latex filled into the mould contains from 0.2 to 2.0 per cent by weight, relative to the latex solids, of an additive selected from sodium salts of ethylene diamine tetraacetic acid, sodium salts of nitrilotriacetic acid and sodium salts of polyphosphates.

The additive is preferably employed in amounts of 0.4 to 1.2 per cent by weight relative to the latex solids.

An expert using the process according to the invention should endeavour to keep the amounts of the specified additive in the latex as low as possible. Whilst with amounts of above about 2.0 per cent by weight the activity of the additive remains preserved, there is such a great increase in viscosity of the latex or of the foam that filling the moulds becomes more difficult, and in the case of complicated moulds, impossible.

The minimum amount required for significant reduction or complete prevention of blistering increases with increasing heat capacity of the mould. Here again the origin of the latex affects the type which can be chosen

[Price 33p]

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and how high a concentration can be chosen. The gelling system which is used can have a further influence. Amounts of the order of magnitude of 0.03 per cent by weight, however, are far from sufficient to produce the desired effect.

The additives for use according to the invention are selected from the sodium salts of ethylenediaminetetraacetic acid and of nitrilotriacetic acid, particularly the tri-sodium salt, and the sodium salts of polyphosphate. An expert can additionally establish, in a few preliminary experiments, for each commercially available latex whether any one of the additives is particularly effective.

In investigations it has been found that Calgon T, Rexene and Rexene NTA are particularly suitable for the following commercially available styrene-butadiene polymer latices: Firestone FRS 247, Hycar 2550 H33 and Pliolite 5358, whilst Calgon T and Rexene NTA are particularly suitable for styrene-butadiene polymer latex Intex E 105. (Calgon, Rexene, Firestone, Hycar, Pliolite and Intex are Registered Trade Marks).

Styrene-butadiene latices are employed for the process according to the invention. The proportion of butadiene units in the polymers lies in the range of 50 to 80% by weight, suitably 65 to 75% by weight, and the proportion of styrene units is 20 to 50% by weight, suitably 25 to 35% by weight.

All latices which are either agglomerated according to a chemical process or are subjected to pressure agglomeration, i.e. are not subjected to freeze agglomeration, are suitable for the process according to the invention.

By mouldings there are understood those latex foam articles which are manufactured in a mould. Carpet backing coatings are therefore excepted from the present process.

Processing of the latices is generally by the Dunlop process. For this, anti-aging agents, sensitisers for gelling, foaming agents, foam stabilisers and vulcanisers are added to the latex, suitably together with the additive characteristic of the process of the invention. This mixture is foamed continuously or discontinuously. After foaming, the gelling agent is added to the latex. The foam is then filled into the moulds. Gelling can take place before or during heating to the vulcanisation temperature.

The vulcanisation takes place in about 30 minutes at approx. 100° C.

By means of the process according to the

invention it is now possible to fill foamed styrene/butadiene latices into hot moulds and to process them further without the undesirable "blistering" occurring. This increases the effectiveness of utilisation of the mould without resulting in significant losses in quality of the foam articles. At the same time, the articles can be manufactured more cheaply.

The following Examples, in which percentages are by weight, illustrate the invention or are comparative.

Examples.

The results summarised in the Table were obtained on carrying out the experiment as follows:

A commercially available styrene/butadiene polymer latex which has not been freeze-agglomerated and contains polymerised styrene and butadiene in a weight ratio between 1:4 and 1:1 (corresponding to 1,000 g of solids) is mixed, whilst stirring, in a foam beater, with 170 g of vulcanisation paste, 58.5 g of potassium oleate (13 per cent strength aqueous solution) as foam stabiliser and the amount of additive indicated in the Table, and foamed to about 12:1. The additives employed in the experiments reported in the Table were the Na₄ and Na₃ salt of ethylenediaminetetraacetic acid (Rexene) and sodium polyphosphate (Calgon T) each experiment being carried out three times, once with each specified additive. After reaching the desired foam height, 100 ml of carboxymethylcellulose (2.5 per cent strength aqueous solution) are stirred into the foam and thereafter about 80 ml of sodium silicofluoride dispersions are added and homogeneously distributed over the course of approx. 2 minutes. The foam is introduced in un-gelled state into the heated mould and the mould is closed and immediately introduced into a steam-heated vulcanisation oven. The vulcanisation time is about 30 minutes. The mouldings are removed from the mould, washed and dried.

Commercially available latices which had been chemically agglomerated and had not undergone freeze agglomeration were used for the experiments. In experiments 1 to 11, a latex with a styrene/butadiene weight ratio of 35/65 and a solids content of about 68% was employed whilst in experiments 12 to 16 a latex with a styrene/butadiene weight ratio of 25/75 and a solids content of approx. 64% was employed.

The vulcanisation paste had the following composition:

	Substance	Parts by weight (g)
5	ZnO	30
	Sulphur	20
	Diphenylguanidine	10
	Zinc diethyldithiocarbamate	10
10	Zinc 2-mercaptobenzothiazole	10
	2,2 - Methylene - bis - (4-methyl - 6 - tert. - butyl-phenol)	5
	Bentonite	0.85
15	Vultamol (Registered Trade Mark) (sodium salt of alkylated naphthalene-sulphonic acid)	4.25
	Water	79.90
		<u>170.00</u>

The sodium silicofluoride dispersion used for gelling had the following composition:

	Parts by weight (g)	
Sodium silicofluoride	25	
Bentonite	2	25
10% strength aqueous potassium hydroxide solution	1	
Water	72	

The mouldings were of size 45 × 45 × 5 cm.

The results obtained with the different additives specified were essentially identical and therefore only one result is quoted for each concentration level. It is representative of the results with all three materials, however.

Examples 1, 6, 9 and 12 in the following Table are comparative.

TABLE

Example No.	Additive (% by weight relative to solids content)	Mould Wall thickness (mm)	Temperature* (°C)	Surface of the foam article
1	0.0	8	65-70	completely covered with blisters
2	0.25	8	65-70	fewer blisters than in 1
3	0.50	8	65-70	occasional minor blisters
4	0.80	8	65-70	free of blisters
5	1.50	8	65-70	free of blisters
6	0.0	4	65-70	smaller and fewer blisters than in Experiment 1
7	0.25	4	65-70	occasional minor blisters
8	0.50	4	65-70	free of blisters
9	0.50	8	80	again increased occurrence of blisters and heat streaks
10	0.50	8	65-70	see Experiment 3
11	0.50	8	50-56	free of blisters
12	0.0	8	65-70	completely covered with blisters
13	0.50	8	65-70	minor areas with blisters
14	0.80	8	65-70	free of blisters
15	1.50	8	65-70	free of blisters
16	0.5	4	65-70	free of blisters

* Temperature of the wall of the mould when introducing the latex.

WHAT WE CLAIM IS:—

1. A process for the manufacture of a latex
foam moulding from a styrene-butadiene poly-
mer latex in which the styrene:butadiene
weight ratio is from 1:4 to 1:1 and which has
not undergone any freeze agglomeration, by
foaming the latex, filling the foam in un-
gelled state into a mould which is at a tem-
perature of from 40 to 70° C. and vulcanis-
ing, wherein the foamed latex filled into the
mould contains from 0.2 to 2.0 per cent by
weight, relative to the latex solids, of an
additive selected from sodium salts of ethylene
diamine tetraacetic acid, sodium salts of
nitrilotriacetic acid and sodium salts of poly-
phosphates.
2. A process as claimed in claim 1, where-
in from 0.4 to 1.2 per cent by weight of
the additive is present in the latex.
3. A process as claimed in claim 1 or 2,
wherein the additive is added to the styrene-
butadiene polymer latex after formation of
the latter but prior to completion of foam
formation.
4. A process as claimed in any of claims

1 to 3, wherein the styrene-butadiene polymer
latex is mixed with a foam stabiliser, vulcan-
iser and additive as specified in claim 1 and
foamed, a gelling agent is added to the foamed
latex, the foam is introduced in un-gelled state
into the hot mould which is then closed and
immediately introduced into a steam-heated
vulcanisation oven.

5. A process as claimed in any of claims
1 to 4, wherein a styrene-butadiene polymer
latex having a styrene-butadiene weight ratio
of from 35:65 to 25:75 is employed.

6. A process for the manufacture of a
latex foam moulding substantially as described
with reference to any of the foregoing Ex-
amples 2 to 5, 7, 8, 10, 11 and 13 to 16.

7. Latex foam mouldings when manufac-
tured by a process as claimed in any of
claims 1 to 6.

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